EXPERIMENTAL INVESTIGATION OF ULTRA-HIGH VACUUM ADHESION AS RELATED TO THE LUNAR SURFACE

SECOND QUARTERLY PROGRESS REPORT

1 OCTOBER THROUGH 31 DECEMBER 1964

N65-20422 (ACCESSION NUMBER)
22 (PAGES)
CR-57631 (NASA CR OR TMX OR AD NUMBER)
1 (THRU)
(CODE)
30 (CATEGORY)
FACILITY FORM 602

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GPO PRICE $ ______
OTS PRICE(S) $ ______

Hard copy (HC) $1.00
Microfiche (MF) $0.50

Prepared for:
NASA/Office of Advanced
Research and Technology
Washington, D. C.

Contract NAS 7-307

Date of Issue:
26 June 1964
A260-BBK2-57

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1.0 INTRODUCTION

This report presents a summary of the work accomplished during the period October through December 1964 on the study of the ultra-high vacuum frictional-adhesional behavior of silicates as related to the lunar surface.

The purpose of this program and the approach used have been detailed in the previous quarterly report. Basically, the purpose is to obtain data relating to the possible behavior of silicates at the lunar surface and to determine the degree to which this behavior can pose problems to lunar surface operations. The approach being used is to obtain quantitative data relating to silicate vacuum friction-adhesion through the use of "single crystal" samples of the various common silicate minerals, and through careful experimental controls. With this approach it is hoped that a basic understanding of the physics of silicate behavior in vacuum can be obtained.

2.0 DISCUSSION

2.1 Vacuum System

In the previous quarterly report it was noted that some questions remained as to whether with the microbalance in the system it would be possible to attain pressures in the low 10^{-10} mm Hg range. This question has now been answered in the affirmative. A number of pumpdowns have been made and in all cases the hoped for low pressure was achieved. With gradual increase in system cleanliness, through use, it may be possible to achieve even lower pressures.

The commercial bakeable valve (separating the low and high vacuum parts of the system) has proved to be a continuing problem with regard to leakage. Only occasionally can a satisfactory closure be made. The valve is closed by forcing a knife edge into an indium gasket. The depth of penetration of the knife edge is controlled by a slip clutch on the valve handle. After a certain number of closures the handle reaches a stop. According to the manufacturer, the indium seat can then be reformed for further use by application of a suitable amount of heat to the outside of the valve. Attempts to improve the valve performance by reforming the gasket have proven unsuccessful. The manufacturer has recommended use of a reflow heater he has developed especially for the valve. We have purchased one of these and will try it. If this does not work we will again contact the manufacturer. To circumvent the problem so that work can continue, we have changed to pinchoff techniques. These have proven moderately successful and have allowed the desired degree of vacuum to be obtained.

One additional vacuum problem arose with regard to use of the high temperature sample outgassing heater. This heater raises the samples to temperatures in the neighborhood of 700-800°C. As noted in the previous report it is outside the vacuum system and sample heating is accomplished by conduction through the chamber wall (see Figure 1). To protect the heater from excessive corrosion during operation, it is immersed in a stream of helium. Unfortunately, it was found that at elevated temperature weld #1 (see Figure 1) leaked badly, raising the system pressure to an intolerably high level. This problem has been overcome by removing welds #1 and #2 through the use of a single machined piece.

Four additional electrical feedthrus have been installed in the system. These are being used to obtain better monitoring of internal temperatures, utilizing thermocouples.

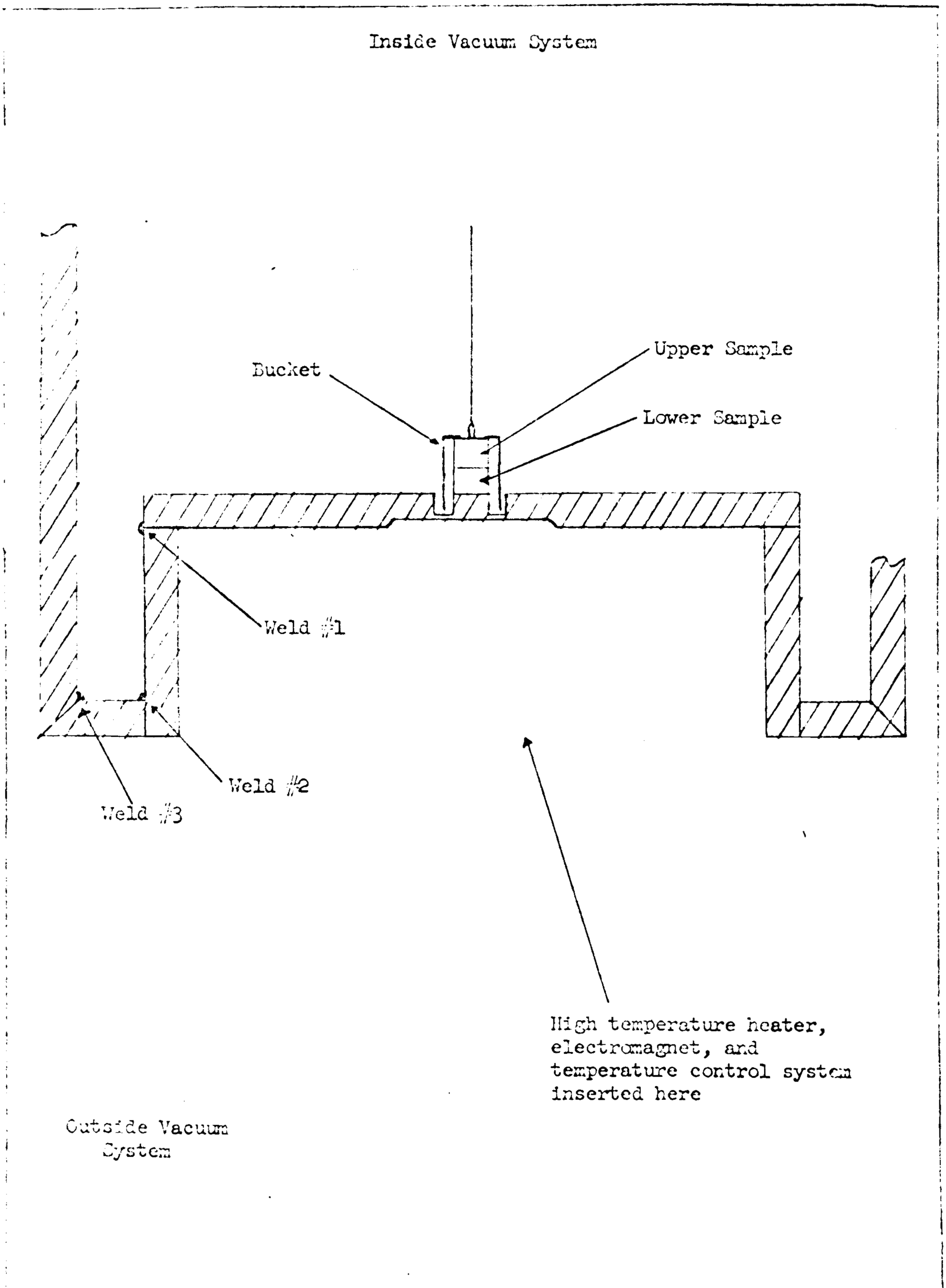

Figure 1 Vacuum System Insert

2.2 The Microbalance

In the previous quarterly report it was noted that difficulties were being experienced in keeping the microbalance zeroed, and that to overcome this difficulty a low melting point counterweight was being tried (the unbalance was found to be in the direction of a decrease in sample weight, so that by suitable application of heat to the counterweight, rebalancing might be obtained). This technique was found to work fairly well, but required an excessive amount of time. It was decided to avoid this problem by utilizing multiple zero lines so that even should drift occur a zero reference line could be found. Theoretically, this makes no difference to the balance calibration. However, in practice some small error may be involved. This has been checked and it has been found that for the actually small amount of drift noted any error present is negligible at the present balance sensitivity.

2.3 Experimental Results

2.3.1 Data

Silicate adhesion has been detected. The data obtained to date are shown in Table I. Three problems are associated with these results. First, for Runs 1-2, the microbalance zero problem had not as yet been resolved. Hence, the possibility exists that some over-contact of the samples occurred, even though great care was taken to avoid this, and therefore the numerical values obtained may in this sense be too high. Second, as noted previously, the high temperature sample outgassing was compromised due to the development of a leak. This not only prevented the desired pressure from being attained, but also negated the effectiveness of the cleaning procedure (it took one day to attain 10^{-9} mm Hg. after the heating and inadvertent admission of helium to the system). This problem has subsequently been

TABLE I

EXPERIMENTAL DATA

(Samples: Orthoclase, 001 faces in contact)

No.	Vacuum Conditions	Surface Cleaning in Vacuo	Applied Load	Sample Orientation	Adhesion Force (μg)	Comments
1.	$\approx 2 \times 10^{-10}$ mm Hg	No high temp. outgassing	None (loading attempts invalidated due to vibration)	$\cong 35^{\circ}$ from atomic match in orientation	$140 \, {}^{+30}_{-10}$ (based on four rdgs.)	Taken prior to soln. of microbalance zero line problem. Vibration problems. Data not reliable and will be discarded.
2.	$\approx 2 \times 10^{-10}$ mm Hg	Same as No. 1	Same as No. 1	$\cong 25^{\circ}$ from atomic match in orientation	320, 340 (based on two rdgs.)	Same as No. 1.
3.	$\sim 8 \times 10^{-10}$ mm Hg.	High temp. outgassing used, but leak developed in weld, compromising attempt	Same as No. 1	$< 1^{\circ}$ from atomic match in orientation	$80 \, {}^{+25}_{-30}$ (based on 22 rdgs.)	Microbalance zero line problem eliminated. Vibration problems remain, hence data not of good quality.
4.	$\approx 8 \times 10^{-10}$ mm Hg	Same as No. 3	Same as No. 1	$\cong 30^{\circ}$ from atomic match in orientation	$50 \, {}^{+20}_{-15}$ (based on 10 rdgs.)	Same as No. 3.
5.	Dry N_2, atmospheric pressure	Brought up to dry N_2 following run No. 4.	Both with and without	Same as No. 4	None detectable ($<$ 10-20 μg)	Air damping eliminated vibration problems
6.	10^{-3} mm Hg	- - - - - - -	Same as No. 5	Same as No. 1	Same as No. 5.	Vibration problems

eliminated. Finally, some degree of vibration was present during all measurements. This was sufficient to invalidate all attempts to measure adhesion after application of load, and to significantly decrease the confidence which can be placed upon the numerical values obtained under zero load. Details of the vibration problem and the techniques used to eliminate it are discussed in a following section.

A few preliminary comments can be made about the data. First, the detection of adhesion forces was definite, when separation occurred it was indicated clearly by an abrupt movement of the microbalance pointer. Also, the sudden parting of the surfaces was visible by direct observation with a cathetometer. Second, the data provide some indication of a crystalline orientation sensitivity. This is by no means conclusive as yet, but if real, provides an important clue as to the nature of the forces acting (discrimination between the various forces which could act is discussed in the following section). Finally, the inability to detect adhesion forces in dry nitrogen (at atmospheric pressure) and at 10^{-3} mm Hg indicates strongly that the adhesion forces acting at UHV are not due to homogeneous electrostatic surface charging. Additional evidence in this regard has been given by attempts, both in vacuum and dry nitrogen, to detect any attractive (or repulsive) forces between the samples as they were slowly brought toward contact. No forces were detected.

2.3.2 Discrimination of Type of Forces Acting

The primary bindings in the silicates are of the ionic-covalent type (e.g. intermediate between the ionic and covalent extremes). The nature and behavior of these bonds have been discussed in the previous quarterly report.

In addition to these, there are forces such as the London - Van der Waals and surface electrostatic which though playing no significant role in silicate bonding can be of importance to investigations of silicate surface adhesion.

Harper (1955) has shown that the contacting of quartz surfaces produces surface electrostatic charges. This can result in a net positive or negative charge. In general, these charges produce long range forces so that if sufficient charging occurs, detectable (by the techniques used in the present experiment) attraction or repulsion can be present, even when the surfaces are not in contact. We shall henceforth call this phenomenon "homogeneous" surface charging as did Overbeek and Sparnaay (1954). Another type of surface charging, denoted as "mosaic charging" has been postulated by Derjaguin (1954). Here the net surface charge may be zero, but positive and negative charge centers are distributed over the surfaces. This type of charge distribution would result in short range adhesive forces. Though there is no completely convincing evidence as to the existence of mosaic charging, particularly for single mineral samples, this must be considered a possible source of adhesive force in the present study.

The London - Van der Waals forces can contribute significantly to silicate adhesion, as evidenced by the work of Bradley (1932), Lowe and Lucas (1953), Jordan (1954), and Derjaguin et al (1954). Though these forces, between two atoms, are quite small and decrease in strength rapidly with atomic separation, they are additive and hence in solid specimens can provide detectable (at least by the techniques used in the present experiment) adhesion. Also of importance is that in solids they are of relatively long range effectiveness, at least as compared to the effective range of the ionic-covalent forces.

It is of interest to consider the methods by which the nature of the measured adhesional forces may be determined. The following techniques are available in the present study:

A. Studies in dry nitrogen or with the inert gases at atmospheric pressure; also studies at moderate vacuum

B. Studies relating to evidence of surface damage produced by adhesion

C. Studies relating to the effects of crystalline orientation (for given faces in contact) upon the adhesion force

D. Studies relating to the load dependence of the adhesion

E. Studies relating to the temperature dependence of the adhesion

F. Studies relating to the mineral dependence of the adhesion

G. Studies relating to surface preparation (roughness)

For Technique A, experience has shown that the short range ionic-covalent forces are not generally effective at these pressures. Hence, only surface electrostatic forces or London Van der Waals forces may be detectable. Should adhesion be detected in UHV but not under the conditions of Technique A, this provides strong evidence that homogeneous surface electrostatic changing is not playing a significant role in the adhesion. It also provides some evidence against the effectiveness of the London - Van der Walls and mosaic forces.

Technique B is one of the most important for determining whether or not the ionic-covalent forces have been brought into play. If evidence of surface disruption (plastic deformation and rupture rather than simple fracturing) after contact is observed, the evidence that the normal bonding forces of the silicate lattice were acting becomes overwhelming, since none of the other forces are sufficiently strong. Unfortunately such effects among the silicates may be difficult to observe due to the general silicate hardness and highly elastic behavior.

Technique C is a very valuable one. Electrostatic forces, whether uniform or mosaic, should not in general be affected by crystalline orientation, particularly for a given crystal face. This is even more true for the London - Van der Waals forces. On the contrary, the magnitude of the ionic-covalent forces should be highly orientation dependent since they are, particularly the covalent bonds, quite directional in nature.

Technique D, if resulting in a load dependent behavior for the adhesion, serves to exclude homogeneous surface electrostatic charges from contributing significantly. Study of the load force-adhesion relations can give information as to which of the others may be primarily responsible.

Technique E, particularly if a temperature dependence exists, can provide evidence as to the elastic-plastic processes acting and hence to the type of forces contributing to the adhesion. Techniques F and G also serve to provide auxiliary information helpful in discriminating between the possible forces.

2.4 Additional Results

2.4.1 Cleaving of Samples

Attempts have been made to cleave those samples possessing good to perfect cleavage planes in a manner so that no surface polishing would be required (this is highly desirable as regards considerations of the nature of the surface layers). The simple device developed for doing this is shown in Figure 2. It consists primarily of a brass cylinder into which two holes have been drilled. The sample is fitted into one of these holes, aligned so that fracture will occur at the proper place and then clamped. A precision machined chisel is inserted in the other hole. To obtain the cleavage fracture the chisel is first placed in contact with the sample and then impacted. Nearly perfect faces have been obtained with this method to date. The few remaining imperfections, consisting of steps, are observable only under high magnification (750x). It appears that with perfection of the technique through practice, faces of suitable flatness can be obtained without polishing.

2.4.2 Surface Dust

The presence of dust on the contacting surfaces can invalidate the adhesion measurements made in this study. Hence, great care must be taken to avoid such contamination. We have developed a technique which minimizes the problem. This involves first applying a light etch to the surfaces. The etch is composed of approximately 30% (by volume) hydrofluoric, 30% glacial acetic, and 40% fuming nitric acid, and has been found effective in removing all particles, as viewed at a magnification of 750x. This is followed by washes with methanol followed by deionized water. The samples are then inserted onto a previously cleaned tube and baked for about one hour at 100°C. Unfortunately, in order to mount the samples in the vacuum system some exposure to the room environment is necessary. It has been found, however, that a light wipe of the sample faces with a chamois cloth, immediately prior to closure of the vacuum system, is generally sufficient to remove any fresh contamination.

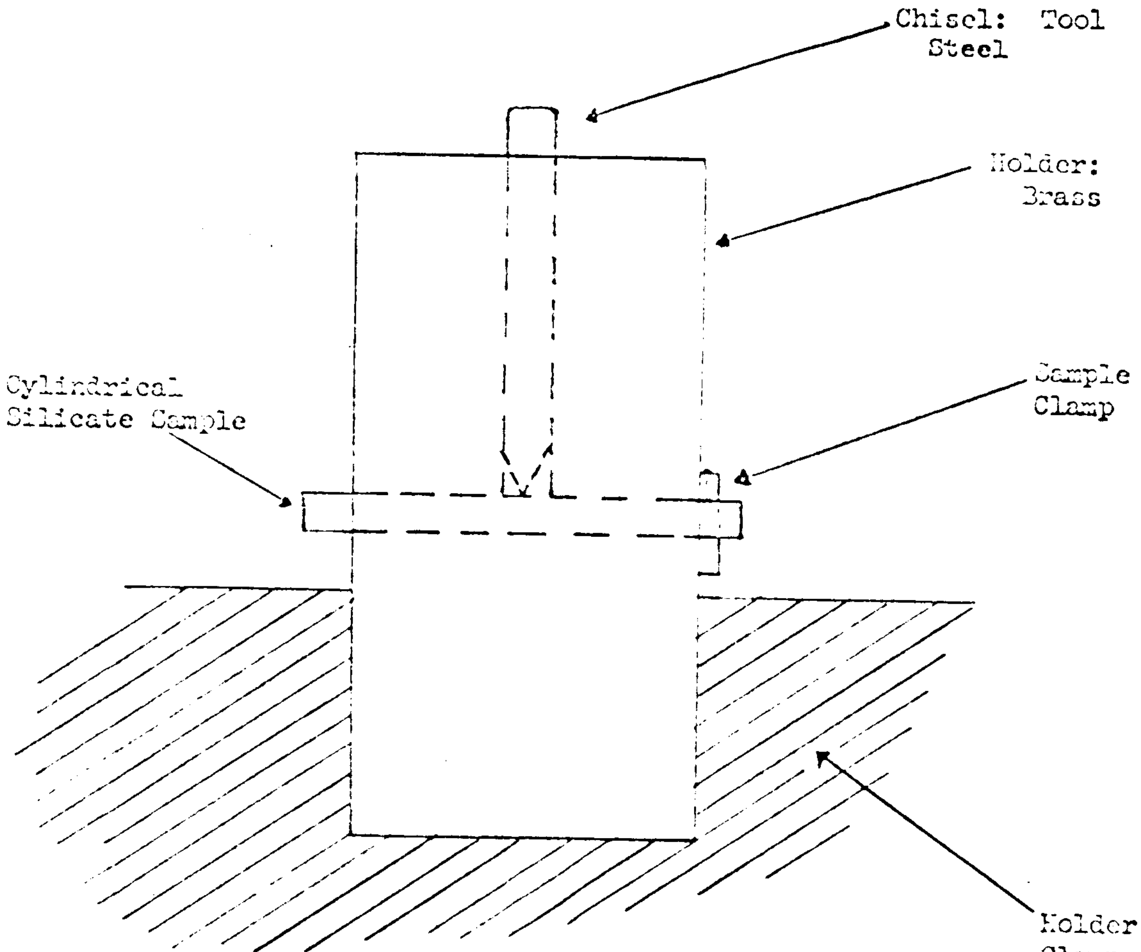

Figure 2 Sample Cleaving Device

2.4.3 Temperature Variation System

The system for varying the sample temperature in the adhesion versus temperature experiments has been built. It is shown in Figure 3, and consists basically of a fluid tank, which is inserted into the same vacuum chamber inset as are the high temperature heater and the electromagnet (see Figure 1). Various temperatures are obtained by pumping particular fluids (liquid nitrogen, refrigerated acetone, heated water etc.) through the tank. Heating and cooling of the samples is by conduction through the chamber walls. Temperature is monitored by means of thermocouples placed inside the vacuum system.

2.5 Vibration Problems and Their Resolution

As noted previously, vibrations passed to the samples from the floor have proven to be a problem. At the beginning of this program it was realized that this could occur. Hence, the vacuum system was mounted on Lord Plateform 200-PM-45 vibration isolators. Tests of this system, in air, showed no sample motion, and it was therefore concluded no vibration problems would be encountered. That this conclusion was erroneous was brought out by the detection of appreciable vibration in vacuum. It appears now that air damping of the microbalance system was sufficient to obscure the vibration at atmospheric pressure.

Since adhesion measurements are compromised by the presence of vibration, it was decided that the vibration isolation must be improved. The vibration - accoustics personnel at Douglas were contacted. They then performed a detailed analysis of the problem. The conclusions reached were:

a. The major problem was due to low frequency vibrations, particularly the vibration components in the horizontal direction. The vibration isolators used did

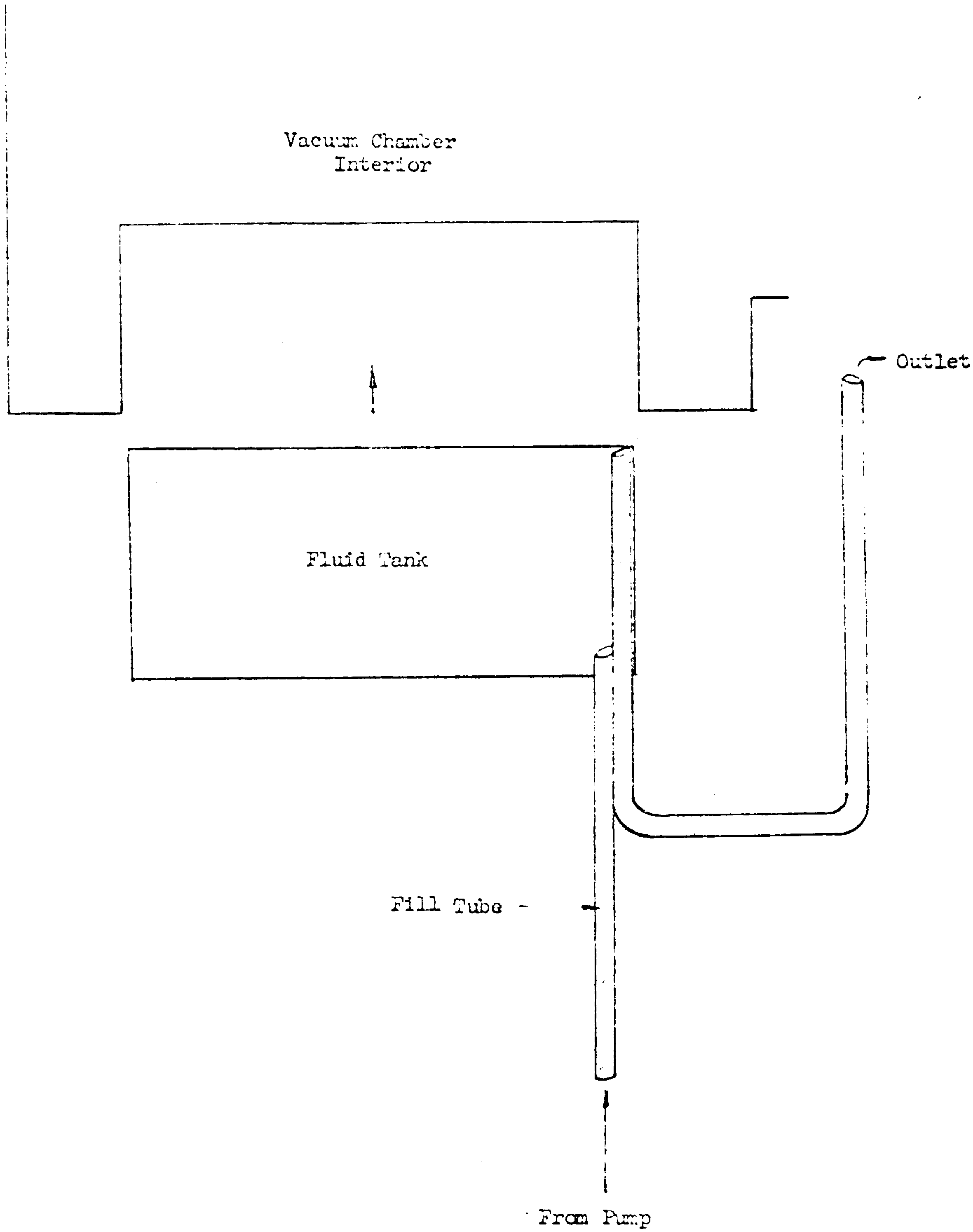

Figure 3. Temperature Control System

not have a sufficiently low natural frequency to damp these out, and were particularly ineffective for the horizontal components.

b. There was no time during the week when floor vibration was sufficiently low not to pose a problem.

c. The isolation system natural frequency must be lowered to about 0.1 - 0.3 cps

d. Of the various vibration isolation techniques available, the best for the present situation is that which involves suspension of the system from "soft" springs. Calculations showed that the springs should be sufficiently soft so as to extend at least 20 inches when loaded.

Details of the studies leading to these conclusions are given in Appendix A. The chosen vibration isolation system is shown in Figure 4. It has been constructed, and the vacuum system will be attached to it shortly.

3.0 SUMMARY

Some adhesion data have been obtained during this quarter. The numerical values obtained are not, however, of high quality. This is due primarily to the presence of vibrations; also to the development of a vacuum system leak during the high temperature outgassing attempt. Both of these problems appear to be resolved, so it is believed that reliable data will be obtained during the following quarter.

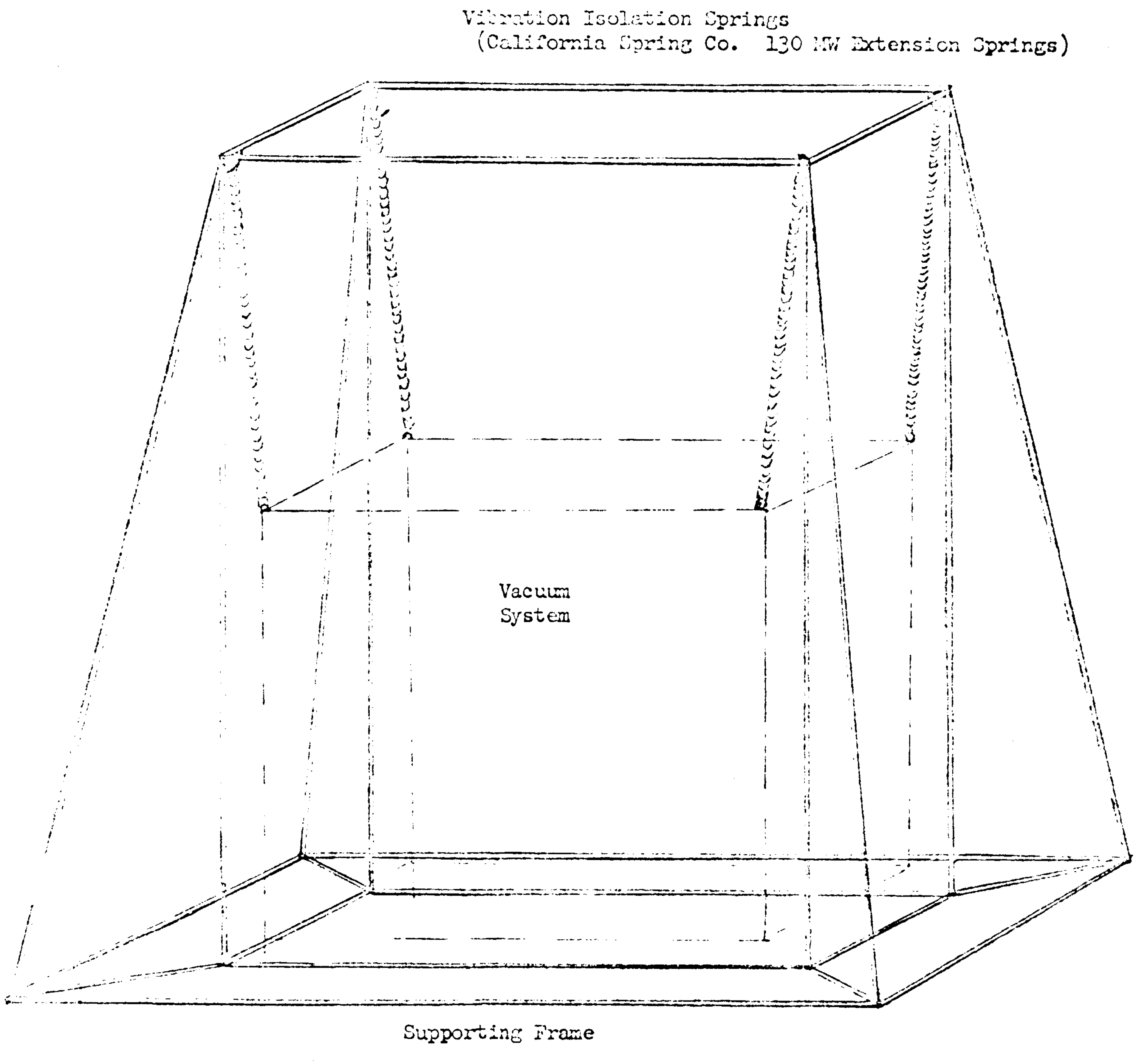

Figure 4. Vibration Isolation System

The data to date are not sufficient for any definitive conclusions to be reached. It does appear, however, that a crystalline orientation dependence of the adhesion may have been detected. If such is indeed the case, it gives strong evidence that the ionic-covalent binding forces are acting. The data taken at atmospheric pressure and at 10^{-3} mm Hg indicate uniform surface charging is not playing a significant role in the adhesion. However, definite conclusions in this regard, as well as to the nature of the force, or combination of forces, acting cannot be made until more and better data are obtained.

APPENDIX A

VIBRATION PROBLEM

The objective of the vacuum adhesion experiment is to measure the adhesion force between two specimens in a vacuum. In order to obtain good measures of this force it is mandatory that very little if any vibration be present. For this reason, the experimental system had been mounted on Lord Plateform 200-PH-45 vibration isolators. These were found to effectively damp out the higher frequencies; however, lower frequencies (a few cps or so) were still present.

In the experiment, one of the samples is suspended from the arm of a microbalance. It was found that in vacuum this specimen, unfortunately, was acting as a pendulum, amplifying ground motions transmitted through the test setup. A lateral pendulum frequency of about 0.75 cps was observed along with a "bounce" or vertical frequency of about 1.5 cps. An approximate analysis showed that the vertical natural frequency of the entire experiment on its mounts was about 7 cps with a "rocking" natural frequency of about 2 cps, and hence that the natural frequency of the vibration isolators being used was not sufficiently low to provide adequate damping. For significant isolation at 0.75 cps, the lateral natural frequency would have to be lowered to about 0.1 - 0.3 cps by using perhaps softer isolators (air bags) or tension springs (or "bungees").

To confirm the diagnosis indicated by the preliminary analysis, a vibration test was performed to measure the natural frequencies of the system. In addition, measurements of the ambient floor and system vibration levels (including spectral distributions) were conducted. The tests indicated that the vertical natural frequency of the experiment on its mounts was actually about 9 cps and the lateral frequency near 2 cps, about as expected. In addition it was found that high frequencies were absent in the system (the plateform mounts were working well in this respect) and that there was no time at which the background noise level was sufficiently low not to be a problem (the background noise level was recorded at ten minute intervals for a period of about 100 hours).

At this point three possible solutions to the problem were considered:

1. Utilize Seismic Mass

   It was thought that the use of a seismic mass, isolated from the building, could provide a more stable location for the experiment. The seismology department at the California Institute of Technology was contacted to determine the required size and type of seismic mass. They suggested that successful utilization of this approach would require data on the dynamic properties of the ground surrounding the mass and, as these data were not available, suggested other approaches be attempted before resorting to a seismic mass.

2. Design and Install an Internal Magnetic Damper on the Pendulum

   If introduction of a magnetic field within the test chamber would not disturb the experiment, addition of a magnetic (eddy current) damper such as is used in sensitive meter movements and chemistry scales could provide the necessary reduction in vibration amplitude. A preliminary design was developed but not used because of questions regarding the effect of the induced magnetic field on the measurements.

3. Design of Lower Frequency Isolation System

   This is the approach that was eventually used and two types of systems were initially considered--air-bags and steel tension springs (stuffed with felt to provide the required damping). The tension springs were selected because they were less expensive and because better data on their characteristics were available. A suspension system as shown in Figure 4 is being utilized as it provides very low lateral natural frequency within a reasonable space.

The vertical natural frequency is given by:

$$f = 3.13 \sqrt{\frac{1}{\delta}}$$

where δ is the static deflection (inches) under a 1 g load. Springs have been selected and fabricated by the California Spring Company which extend 30 inches under the rated load (extended length of 50 inches total) providing a vertical natural frequency of less than 0.6 cps.

The lateral natural frequency is that of a pendulum. The arm is the 50 inch spring plus the distance from the spring attachment to the c.g. (estimate 20 inches).

$$f = \frac{1}{2\pi} \sqrt{\frac{g}{l}}$$

l = the length of the arm (in.)

g is the gravitation constant (386 in/sec)

For a 70 inch pendulum $f < 0.4$ cps and the resulting vibration of the specimen should be considerably reduced from its present value.

The "A" frames and tension springs are currently being assembled and will be completed and evaluated early in January.

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